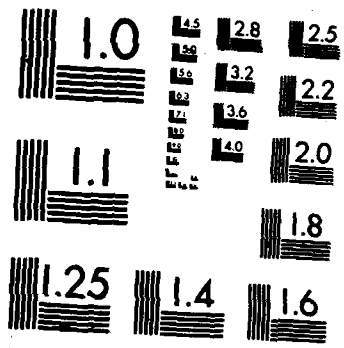
AD-A174 530 ULTRA-LOW THERMAL EXPANSION CERAMICS(U) PENNSYLVANIA STATE UNIV UNIVERSITY PARK MATERIALS RESEARCH LAB H A MCKINSTRY ET AL AUG 86 AFOSR-TR-86-2060 1/1

UNCLASSIFIED AFOSR-83-0291 F/G 11/2 NL

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NATIONAL BUREAU OF STANDARDS-1963-A

ULTRA-LOW THERMAL EXPANSION CERAMICS

AD-A174 530

AFOSR-TR- 86-2060

Final Scientific Report

Submitted to:

Department of the Air Force
AFOSR/NC
Contract No. AFOSR-83-0291

Authors:

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August 1986

MATERIALS RESEARCH LABORATORY

THE PENNSYLVANIA STATE UNIVERSITY

UNIVERSITY PARK, PENNSYLVANIA 16802

UNCLASSIFIED
SECURITY CLASSIFICATION OF THIS PAGE

AD-A 174530

# REPORT DOCUMENTATION PAGE

**1a. REPORT SECURITY CLASSIFICATION**

**1b. RESTRICTIVE MARKINGS**

**2a. SECURITY CLASSIFICATION AUTHORITY**

**2b. DECLASSIFICATION/DOWNGRADING SCHEDULE**

**3. DISTRIBUTION/AVAILABILITY OF REPORT**
Approved for public release, distribution unlimited

**4. PERFORMING ORGANIZATION REPORT NUMBER(S)**

**5. MONITORING ORGANIZATION REPORT NUMBER(S)**
AFOSR-TR- 86-2060

**6a. NAME OF PERFORMING ORGANIZATION**
The Pennsylvania State University

**6b. OFFICE SYMBOL** *(If applicable)*

**7a. NAME OF MONITORING ORGANIZATION**
AFOSR

**6c. ADDRESS** *(City, State and ZIP Code)*
201 Materials Research Laboratory
University Park, PA 16802

**7b. ADDRESS** *(City, State and ZIP Code)*
Bldg. 410
Bolling AFB, DC 20332

**8a. NAME OF FUNDING/SPONSORING ORGANIZATION**
Dept. of the Air Force

**8b. OFFICE SYMBOL** *(If applicable)*
NC

**9. PROCUREMENT INSTRUMENT IDENTIFICATION NUMBER**
AFOSR-83-0291

**8c. ADDRESS** *(City, State and ZIP Code)*
Air Force Office of Scientific Research
AFOSR/NC Bldg. 410
Bolling AFB, DC 20332

**10. SOURCE OF FUNDING NOS.**

| PROGRAM ELEMENT NO. | PROJECT NO. | TASK NO. | WORK UNIT NO. |
|---|---|---|---|
| 61102F | 2303 | A3 | |

**11. TITLE** *(Include Security Classification)*
Ultra-low Thermal Expansion Ceramics

**12. PERSONAL AUTHOR(S)**
H.A. McKinstry, D.K. Agrawal, G.E. Lenain, C.S. Vikram, S.Y. Limaye, A. Patankar

**13a. TYPE OF REPORT**
Final Report

**13b. TIME COVERED**
FROM 7/83 TO 6/86

**14. DATE OF REPORT** *(Yr., Mo., Day)*
August 1986

**15. PAGE COUNT**

**16. SUPPLEMENTARY NOTATION**

**17. COSATI CODES**

| FIELD | GROUP | SUB. GR. |
|---|---|---|
| | | |
| | | |

**18. SUBJECT TERMS** *(Continue on reverse if necessary and identify by block number)*
Low Thermal Expansion [NZP]-Family, Laser Speckle Dilatometer, X-ray Diffraction Thermal Expansion, Thermal Expansion of Perovskites.

**19. ABSTRACT** *(Continue on reverse if necessary and identify by block number)* M superscript I   M superscript II


The crystal chemistry, ~~synthesis~~, and thermal expansion investigation of [NZP]-family was completed--in that $M^IZr_2P_3O_{12}$ ($M^I$ = Li, Na, K, Rb, Cs), $M^{II}Zr_4P_6O_{24}$ ($M^{II}$ = Mg, Ca, Sr, Ba) and a few more compositions were studied. Single crystals of [NZP] and [CZP] were grown, and high-temperature x-ray measurements were made on these crystals to test the validity of a structural model to interpret thermal expansion data. The possibility of the development of a glass-ceramic of $NaGe_2P_3O_{12}$ was explored. Dielectric measurements made on $CaZr_4P_6O_{24}$ reveal that in general [NZP]-materials have a low dielectric constant.

alpha

Three new families, namely diborides ($ZrB_2$, $TiB_2$, and $CrB_2$), $Al_2O_3$-$GeO_2$ system and perovskite $Pb(Mg_{1/3}Nb_{2/3})O_3$ were investigated in order to search for any new low thermal expansion composition, but not much success was achieved in this direction, except that some perovskite compositions displayed low $\alpha$ behavior at low temperatures.


**20. DISTRIBUTION/AVAILABILITY OF ABSTRACT**
UNCLASSIFIED/UNLIMITED ☐ SAME AS RPT. ☐ DTIC USERS ☐

**21. ABSTRACT SECURITY CLASSIFICATION**

**22a. NAME OF RESPONSIBLE INDIVIDUAL**
Dr. Ulrich

**22b. TELEPHONE NUMBER** *(Include Area Code)*
767-4963

**22c. OFFICE SYMBOL**
NC

DD FORM 1473, 83 APR — EDITION OF 1 JAN 73 IS OBSOLETE.

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SECURITY CLASSIFICATION OF THIS PAGE

New magnetic materials with low thermal expansion characteristics were developed by (i) making ionic substitution in [NZP]-structure and (ii) by adopting a diphasic composite approach. Such a material was produced using $Na_4Zr_2Si_3O_{12}$ and $Y_3Fe_5O_{12}$.

A di/multiphasic approach was adopted to tailor and control the thermal expansion of a ceramic, in this approach [NZP] was mixed with a second phase which is thermodynamically compatible. The sample was fabricated by a suitable heat treatment. Several compositions which demonstrate very low α over a wide temperature range were produced.

Finally, several new techniques for measuring ultra-low thermal expansion with high precision and accuracy utilizing laser spectroscopy were developed.

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## SUMMARY


I. Synthesis, crystal chemistry, and thermal expansion of [NZP]-family of materials: The work on [NZP] or [CTP] family can be subdivided into the following areas:

1. Synthesis and thermal expansion of $M^{I}A_2P_3O_{12}$ ($M^{I}$ = Li, Na, K, Rb, Cs) and $M^{II}A_4P_6O_{24}$ ($M^{II}$ = Mg, Ca, Sr, Ba) where A stands for Zr and Ti.
2. Single crystal growth of $CaZr_4P_6O_{24}$ and $NaZr_2P_3O_{12}$ by flux method.
3. Development of a glass-ceramic with [NZP]-composition.
4. Dielectric studies of $CaZr_4P_6O_{24}$.
5. Investigation of crystalline constraints on thermal expansion of $NaZr_2P_3O_{12}$.
6. Exploratory substitution in the [NZP]-structure to find any new low $\alpha$ compounds.

II. Exploration for new families of low-expansion materials: The following new families for possible low $\alpha$ characteristics were investigated:

1. Diborides ($ZrB_2$, $TiB_2$, $CrB_2$, $MnB_2$ and $YB_2$).
2. $Al_2O_3$-$GeO_2$ system.
3. Pb $(Mg_{1/3}Nb_{2/3})O_3$ and related perovskites.

III. New low thermal expansion magnetic materials:

1. Mono-phasic magnetic [NZP]-compositions.
2. Diphasic composite approach to produce low $\alpha$ ceramics with ferrimagnetic properties.

IV. Development of di/multiphasic micro-composite ceramics for low $\alpha$ applications: The systems studied include [NZP] or [CZP]+$Nb_2O_5$, $ZrSiO_4$, $GdPO_4$, $Mg_3(PO_4)_2$, $Zn_3(PO_4)_2$, MgO and ZnO.

V. New methods for measuring ultra-low thermal expansion: In this area, with the aid of laser technology, several new techniques for the measurements of very low $\alpha$ of ceramic materials were developed.

FINAL REPORT

During the three years (July 1983-June 1986) of the contract period of the project entitled, "Ultra-low Thermal Expansion Ceramics," most of the research work stated in the original proposal has been successfully completed. In this final report, a summarized version of the past three annual reports in a cumulative form is being presented.

## I. Synthesis, Crystal Chemistry, and Thermal Expansion of [NZP]-Family

In this area, several aspects of research include synthesis, characterization, thermal expansion, single crystal growth, crystal chemistry, development of a glass-ceramic, dielectric properties, etc. It was established (1) that the sol-gel process is superior to the solid state reaction method (powder mixing) for synthesizing most of the [NZP]-family members. Further, the traditional sol-gel technique was modified by introducing a seeding step which has improved considerably the sinterability and the microstructure of the material. Single crystals of $CaZr_4P_6O_{24}$ and $NaZr_2P_3O_{12}$ were grown by a flux technique, and measurements were made on the crystals supplied by Perrotta (ALCOA); (2) by using facilities at Geophysical Laboratories, Washington, DC. A systematic investigation of the thermal expansion of alkaline [NZP] ($M^{I}Zr_2P_3O_{12}$ where $M^{I}$ = Li, Na, K, Rb, Cs) and alkaline earth [NZP] ($M^{II} = Zr_4P_6O_{24}$ where $M^{II}$ = Mg, Ca, Sr, Ba) was completed (3,4). A structural model (5) to interpret the thermal expansion data in these systems and based on structural parameters of [NZP], was developed; this model can explain the anisotropic thermal expansion of other [NZP]-family members.

It has been shown that $CaZr_4P_6O_{24}$ (CaZP) and $SrZr_4P_6O_{24}$ (SrZP) which are structurally isomorphous with $NaZr_2P_3O_{12}$ [NZP] have low bulk thermal expansion, in fact, CaZP exhibits low negative CTE (coefficient of thermal expansion, $\alpha$)

and SrZP low positive α between 25° and 500°C. However, a new discovery about these materials is that the anisotropies in their axial thermal expansions are reversed, i.e., in CaZP c-axis expands and a-axis contracts while on the other hand the SrZP c-axis contract and a-axis expands. This has been demonstrated in Figure 1 also, in which the lattice parameters 'a' and 'c' have been plotted against temperature. The lattice parameters were determined using powdered material and high temperature X-ray diffractometry. In order to control the thermal expansion properties of a crystalline ceramic material, it is essential that the material exhibits both a low net coefficient of thermal expansion, and a low anisotropy of thermal expansion. The latter is very important for controlling thermal shock in sintered ceramics. We have reduced this to practice and show (see Figure 2) that the anisotropy of α at $Ca_{0.5}Sr_{0.5}Zr_4P_6O_{24}$ is in fact very close to zero. This is an important discovery, a U.S. patent has been filed for this work.

Several compositions in the [NZP]-family were considered for the development of a glass-ceramic and finally, $NaGe_2P_3O_{12}$ because of its low melting temperature (∿ 1125°C), was found suitable for this purpose (6). Dielectric measurements made on CZP+ MgO, ZnO system revealed that, in general, [NZP] materials have low dielectric constants with relatively low loss.

The crystal chemistry of [NZP] or [CTP]-family (7) was investigated by making numerous substitutions at various lattice sites of [NZP]-structure and also with the aid of published data on this subject. The [NZP]-structural family has an extraordinary range of discrete compositions and crystalline solutions, these compositions being classified according to their crystal chemical substitution schemes.

The effect of polycrystal constraints on the thermal expansion of a material was studied using a powdered and sintered sample of $NaZr_2P_3O_{12}$ (8).

## II. Exploration of New Families of Low Expansion Materials

In this area, three different systems, namely diborides, study the anisotropy $Al_2O_3$-$GeO_2$ and $Pb(Mg_{1/3}Nb_{2/3})O_3$--perovskite families, were studies in order to look for any new low or near zero expansion compositions. Several diborides, namely $ZrB_2$, $TiB_2$, $CrB_2$, $MnB_2$ and $YB_2$, were chosen, and their axial thermal expansions (9) were measured by high temperature X-ray diffractometry to study the anisotropy in thermal expansion. Several compositions in $Al_2O_3$-$GeO_2$ were synthesized by a powder mixing technique, and their thermal expansions were determined using a Harrop Dilatometric Analyzer. In the perovskite family, synthesis and thermal expansion property measurements of several new compositions such as PMN, modified PMN (10), PZT, and their solid solutions were done. Most of the compositions exhibited large thermal expansions; however, some PMN and PZN based compounds demonstrated very low $\alpha$ at low temperatures.

## III. New Low Thermal Expansion Magnetic Materials

Two approaches were adopted to produce new magnetic materials with low thermal expansion characteristics: (i) development of mono-phasic [NZP] compositions by incorporating rare-earth ions ($Y^{3+}$, $Gd^{3+}$), $Fe^{3+}$ and $Cr^{3+}$ at Na, Zr, or P sites. Only $Cr^{3+}$ could be substituted for Zr with excess Na or Ca (11) for charge compensation; (ii) using the diphasic approach in which a suitable [NZP]-compound with negative $\alpha$ is mixed with a thermodynamically compatible magnetic material, and a ceramic is fabricated by suitable heat treatment. Development of such a ceramic with 85% $Na_4Zr_2Si_3O_{12}$ + 15% YIG (Yttrium Iron Garnet) which displays very low thermal expansion up to 150°C (12) has been successful.

## IV. Development of Di/Multiphasic Micro-composite Ceramics for Low $\alpha$ Applications

The diphasic micro-composite approach provides an avenue for more firmly

controlled "tailoring" of properties of the materials. The desirable properties of two different materials can be brought together. For this purpose, the specific systems studied include [NZP] or [CZP]+$Nb_2O_3$, $GdPO_4$, $ZrSiO_4$, $Mg_3(PO_4)_2$, $Zn_3(PO_4)_2$, MgO and ZnO. Several compositions exhibited a near zero expansion profile over a wide temperature range (13).

## V. New Methods for Measuring Ultra-low Thermal Expansion

Since the development of a new class of materials with ultra-low thermal expansion, the conventional push-rod dilatometers have become obsolete due to their limitations in measuring precisely, and with high accuracy, very low thermal expansion behavior. To resolve this problem, several new measuring techniques (14-17) utilizing laser spectroscopy have been developed in the past three years. These techniques are highly sensitive, precise, and furnish the data with high accuracy. They are capable of measuring ultra-low thermal expansion of ceramic materials.

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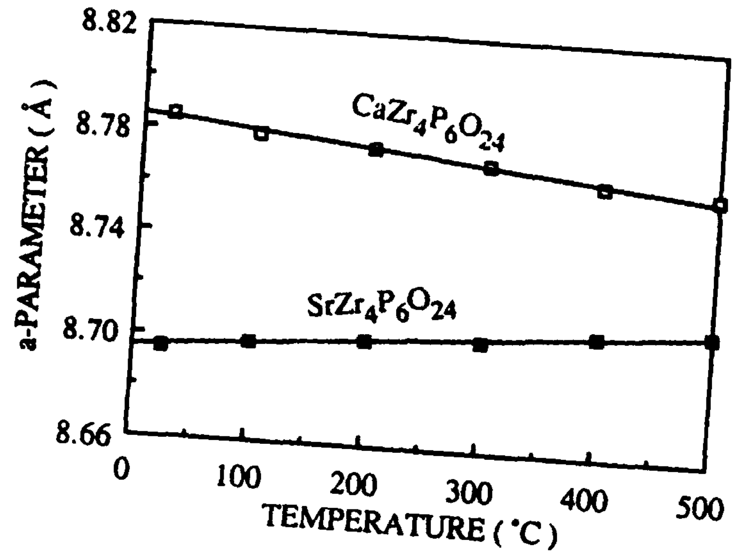
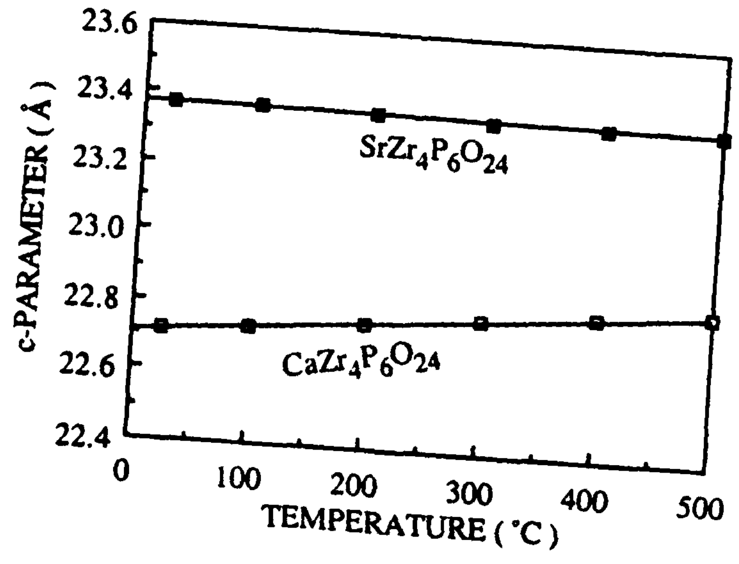
**AXIAL THERMAL EXPANSION $CaZr_4P_6O_{24}$ AND $SrZr_4P_6O_{24}$**



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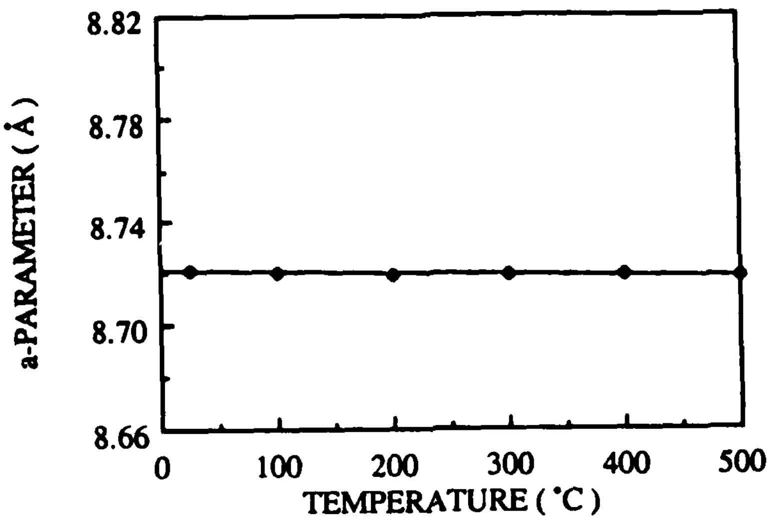


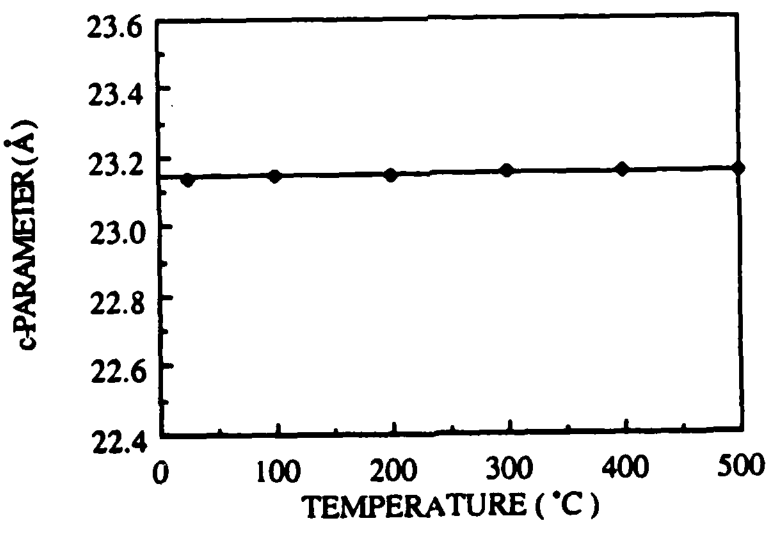

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